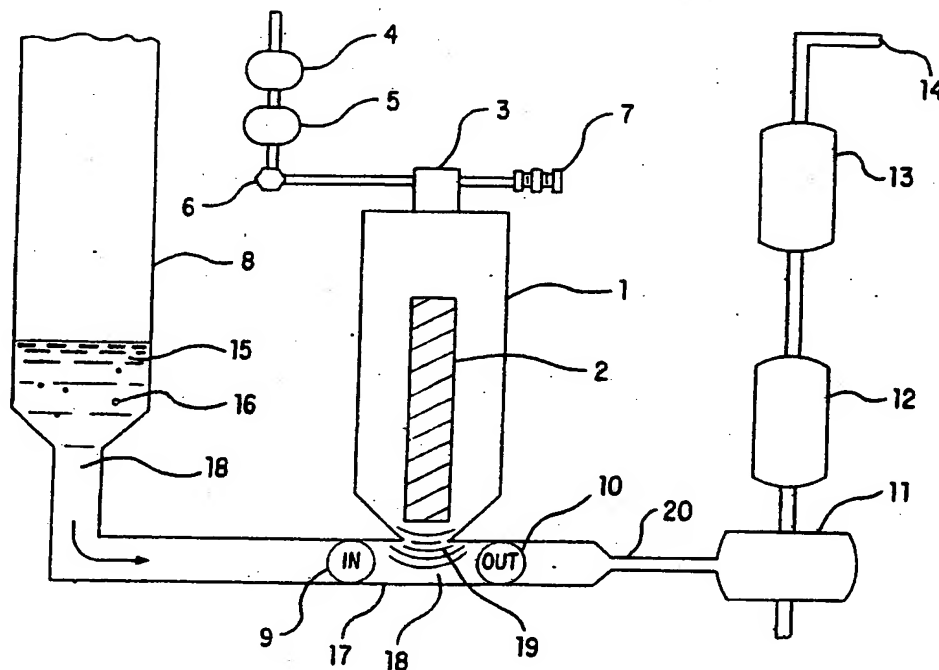




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : B02C 23/36		A1	(11) International Publication Number: WO 92/09368
			(43) International Publication Date: 11 June 1992 (11.06.92)
(21) International Application Number: PCT/US91/09089		(74) Agent: LIPTON, Robert, S.; Lipton & Famiglio, P.O. Box 546, Media, PA 19063 (US).	
(22) International Filing Date: 3 December 1991 (03.12.91)			
(30) Priority data: 623,106 3 December 1990 (03.12.90) US		(81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), MC (European patent), NL (European patent), SE (European patent), SU ⁺ , US.	
(60) Parent Application or Grant (63) Related by Continuation US 623,106 (CIP) Filed on 3 December 1990 (03.12.90)			
(71)(72) Applicant and Inventor: REDDING, Bruce, K., Jr. [US / US]; 2708 S. 86th Street, Philadelphia, PA 19153 (US).		<p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	

(54) Title: APPARATUS AND METHOD FOR MICRONIZING PARTICLES



(57) Abstract

This invention includes an apparatus and method for the reduction in size of particulate matter (16). The particles (16) are dispersed within a fluid medium (15). At least one pressure pulse (19) is used to cause a particle (16) to break up into two or more smaller-sized resultant particles (16B). The resultant particles (16B) are then filtered or otherwise separated from the fluid medium (15).

BEST AVAILABLE COPY

+ DESIGNATIONS OF "SU"

Any designation of "SU" has effect in the Russian Federation. It is not yet known whether any such designation has effect in other States of the former Soviet Union.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MG	Madagascar
AU	Australia	FI	Finland	ML	Mali
BB	Barbados	FR	France	MN	Mongolia
BE	Belgium	GA	Gabon	MR	Mauritania
BF	Burkina Faso	GB	United Kingdom	MW	Malawi
BG	Bulgaria	GN	Guinea	NL	Netherlands
BJ	Benin	GR	Greece	NO	Norway
BR	Brazil	HU	Hungary	PL	Poland
CA	Canada	IT	Italy	RO	Romania
CF	Central African Republic	JP	Japan	SD	Sudan
CG	Congo	KP	Democratic People's Republic of Korea	SE	Sweden
CH	Switzerland			SN	Senegal
CI	Côte d'Ivoire	KR	Republic of Korea	SU+	Soviet Union
CM	Cameroon	LI	Liechtenstein	TD	Chad
CS	Czechoslovakia	LK	Sri Lanka	TC	Togo
DE	Germany	LU	Luxembourg	US	United States of America
DK	Denmark	MC	Monaco		

APPARATUS AND METHOD FOR MICRONIZING PARTICLES

Technical Field

This invention generally relates to the field of reducing the size of particles and, particularly, to reducing the size of particles which are dispersed within a liquid medium.

Background Art

Micronizing, the process of reducing the size of particles, has many applications, such as in the manufacture of pharmaceuticals, pesticides, paints, adhesives, and many other chemical products. As is well-known in the prior art, micronizing can be accomplished by several well-known means. Solid particulates may be mixed with a liquid carrier, or medium, and an abrupt force may be applied to the dispersion to cause size reduction. Alternatively, solid particles may be mixed with a solvent, permitting them to be dissolved. The solvent is then removed, generally by evaporation. The resultant residue usually consists of particles which are smaller than the original particles.

Another prior art method includes the application of cryogenic temperatures to particles through the use of liquid nitrogen, for example. The particles either spontaneously fracture or become more easily fractured into smaller pieces. In probably the oldest method, particles are simply ground between two surfaces, breaking them into smaller ones. Here, a direct mechanical force may be applied by an anvil or hammer to the material to be reduced in size.

Each of these methods is effective, but often there is low efficiency in size reduction. Frequently, the resulting size range of the particles is non-uniform and overly broad. Furthermore, the prior art methods are less effective with harder substances which tend to have a greater resistance to size reduction. Many prior art methods are of the batch type which use non-continuous size-reduction processes having low flow rates, high energy and economic costs and material handling problems.

European Patent application 79302842.4, filed 10 December 1979, teaches a continuous flow process. Particles in a carrier medium are passed between a series of opposing pistons which impart a shock to the

particles, causing them to break into smaller ones. The process taught uses as many as 50 pairs of plungers.

Disclosure of Invention

This invention seeks to overcome the problems with the
5 conventional technologies for particle size reduction through a novel method of applying pressure pulses to impart shear forces on the subject particles.

This invention employs a dispersion of particles in a liquid medium, or slurry, which, when exposed to a pressure pulse, results in the fracture
10 of the particles. While some fractured particles disassociate completely into smaller sub-particles or fragments, other particles do not break apart, but instead merely crack along a fracture interface. The dispersed particles within the liquid medium may then be caused to flow through a series of baffles, within a flow tube, which generate shear forces and
15 turbulence. This causes those fractured particles which have not completely broken apart to do so.

In its simplest form, the present invention is an apparatus and method for reducing the size of particles dispersed within a fluid medium. Small portions of the dispersion are subjected to an abrupt pressure
20 change or a series of abrupt pressure changes through the use of a modified hydraulic piston pump. It has been found that the application of the abrupt pressure to the dispersion acts to fracture the dispersed solids, almost instantly, into smaller pieces. The micronized particles and the liquid medium are evacuated from the pump compressor chamber before
25 the next load of the dispersion is received.

The compression chamber which permits the application of high pressures has a pair of valves, an input and an output valve. The valves cooperate to confine a portion of the dispersion in the compression chamber during the application of the abrupt pressure change. After the
30 portion has been treated, the valves permit successive portions of the dispersion to be processed. Although the process is literally a batch process, the small batches are treated so rapidly that, from an operational

point of view, the process is functionally a continuous process with all of its advantages. Of course, under some circumstances, a batch process may be desired.

The resulting particles formed may have been reduced to the desired size after one application of the process, or they may require additional passes through the compression chamber. As previously mentioned, in some cases the particles are fractured to the extent desired, but they do not actually separate into smaller particles. In this case, separation may be effected through the use of baffles within a tube which act to increase the shearing forces and turbulence applied to the dispersion as it flows through the tube. The additional turbulence causes high shear to act upon those particles which were not completely fractured by the pressure pulse treatment, resulting in a further size reduction of the dispersed particles. One or more filter screens may be placed within the flow tube which act to further fracture the particles as they are rammed at high velocity and higher pressure against the screens.

The present invention may also be used to complete the size reduction process begun by a conventional technique.

It is, therefore, a primary object of the present invention to provide an apparatus and method for breaking particles into smaller particles by applying an abrupt pressure change to particles dispersed within a liquid carrier medium.

It is a further object of the invention to provide an apparatus and method for effecting the reduction of the size of particles which are dispersed within a liquid medium by applying an abrupt pressure change to the dispersion while it is contained in a pressure chamber.

It is another object of the invention, in accordance with the preceding objects, to apply the abrupt pressure change by a piston or plunger.

It is another object of the invention to provide an apparatus and method, in accordance with the prior objects, wherein an intermittent flow of the dispersion is provided to the compression chamber.

It is still another object to provide an apparatus and method to reduce the size of particles, wherein both the average size of the particles and the narrowness of the size range can be easily adjusted.

Other objects and advantages of the invention will be apparent to persons skilled in the art, from a reading of the following "Brief Description of the Drawings," the "Detailed Description of the Invention," and the appended claims.

Brief Description of the Drawings

Figure 1 is a schematic diagram of the micronizing apparatus.

Figure 2 is an illustration of the initial effect of the apparatus device on a target particle.

Figure 3 is a schematic of the baffle chamber which may be used to assist in completing the micronizing of target particles.

Figure 4 is a schematic of the pump used in creating the abrupt pressure change and the spring-loaded locking check valves used in the pressure chamber.

Figure 5 is a schematic of the pump-operated pressure size reduction apparatus in combination with the filter and size chamber attachments connected.

Detailed Description of the Invention

The present invention is an apparatus and method for reducing the size of particles which use an abrupt pressure change to effect the break up or fracturing of particulates dispersed within a liquid medium.

Referring to Figure 1 where the preferred embodiment of the invention is illustrated, a dispersion or slurry 18 comprised of solid or semi-solid particles 16 dispersed in a liquid medium 15 is drawn from a reservoir 8 into a piston pressure treatment apparatus of the present invention which includes a hydraulic pump 1. The slurry 18 is drawn into the compression chamber 17 of the pump 1, as the piston 2 is raised. In another embodiment of the present invention, the dispersion can be injected into the compression chamber under pressure.

The pump 1, of the preferred embodiment of the invention, is an air-operated hydraulic pump with a single piston which is used to generate the abrupt pressure change. Alternatively, a motor which is either electrically, pneumatically or mechanically driven may also be used to drive a piston. Additionally, multiple pistons may be used instead of the single piston illustrated in Figure 1. The apparatus illustrated in Figure 1 is a modified air-operated hydraulic pump with an air motor 3 which is powered by compressed air which enters air motor 3 by way of a pressure regulator 4 and oiler device 5, through a 1/4 turn air valve 6. Exhaust is vented through a muffler 7 to reduce noise. The regulator 4 controls the pressure level of the air input. The 1/4 turn valve 6 controls the rate of air flow, which correspondingly controls the stroke count of the piston 2, increasing or decreasing the speed of the pump 1.

Slurry 18 is drawn past inlet valve 9 and deposited in the compression chamber 17 of the invention by action of the piston 2. When the piston is raised it draws slurry 18 into the compression chamber 17. In a normal hydraulic pump, acting as a pump, a liquid would be expelled from the pump as the piston is moved downward, with the inlet valve closing and the outlet valve opening to allow the liquid to be pushed from the chamber. In the present invention, however, the method of operation is changed so that as the piston 2 moves downward, both the inlet 9 and the outlet valve 10 are locked in the closed position for a period of time. The piston 2 impacts with the slurry 18 and generates an abrupt pressure change which causes a shock wave or pressure pulse 19 to pass through the slurry 18. The pressure pulse 19 is of such intensity as to cause the particles 16 dispersed within the liquid medium 15 to become broken, fractured or crushed into smaller-sized bit particles. More than one stroke of the piston may, in the alternative, be implemented to increase particle size reduction.

Eventually, outlet valve 10 opens. The pressure built up within the compression chamber 17 forces the pressure-treated slurry containing the fractured particles to exit the compression chamber 17. The flow carries

the treated slurry into a block valve 11. The flow channel or tube 20 between the output valve 10 and the block valve 11 has a reduced diameter from the channel or tube exiting from the output valve 10. This causes a back pressure to act upon the output valve 10 helping to delay its opening, and thus helping to adjust the timing of the operation of the valves which is necessary for proper pressure pulse generation.

The power applied by the piston 2 to the slurry dispersion 18 depends on the time interval during which the piston 2 is acting upon the slurry 18. The pressure pulse intensity is determined by the amount of pressure fed into the air motor 3. In most hydraulic pump assemblies, the geometry of the pump design will multiply the effective pressure, the instantaneous pressure present within the compression chamber, by a factor of 100 to 200 times. The apparatus used in the experiments of the present invention had a multiplier of 110 times the gauge inlet pressure setting. Therefore, a pressure gauge reading of 60 psi inlet pressure is actually 6,600 psi effective within the compression chamber.

The time interval during which the pressure is applied by the pump, the period during which the slurry dispersion is exposed to high pressure, is determined by the cycle time of the piston 2 and the timing of the opening of the inlet and outlet valves 9 and 10. By adjusting the timing of the valves 9 and 10, the particles within the slurry are exposed to high pressure for a longer period of time, allowing the pressure to work longer toward fracturing the particles.

By adjusting the stroke speed and the function of valve openings, it is possible to fracture some of the hardest substances. The pressure treatment factors will vary for different particulate materials.

The pump and piston used to pressurize the dispersion is also used to move the dispersion through the system. The pump draws the pre-dispersion continuously through the system, applies the abrupt pressure change, and then pushes the pressure-treated dispersion through the remainder of the system.

If the pressure exerted by the piston 2 during its compressive stroke

is increased, the system tends to form smaller particles. If the pressure setting is decreased, the system tends to reduce the particulate size only slightly. Repeated piston strokes at a constant pressure setting also increases particle size reduction.

5 Particulate which are fractured by the piston device, as illustrated in Figure 2, may not fully separate or break apart into disassociated particles of smaller size. The pressure fractionizing process taking place within the compression chamber 17 of the pressure treatment device 1 is shown in Figure 2. The piston 2, on its downward stroke, acts to generate a
10 pressure pulse wave 19 which meets a particle which has been dispersed within the liquid carrier medium 15 and causes that particle to fracture, resulting in fractured particles 16B.

 The pressure pulse wave 19 is generated by the action of the piston 2 as it makes contact with the liquid medium 15 while both the inlet and
15 outlet valves 9 and 10 are closed, thus locking the slurry within the compression chamber 17. The base of the compression chamber 17B also plays an important role in that its shape and geometry can aid in developing echo effects within the compression chamber which may enhance fracturization of the particle. While only one particle is shown in
20 Figure 2 for purposes of clarity, it is obvious that many particles are simultaneously treated by the invention.

 Eventually, outlet valve 10 opens and residual pressure forces the slurry to enter into additional treatment chambers, be recycled through the device, or be evacuated entirely. The pressure pulse acting on particles
25 16 may be powerful enough to complete the fracturing, and therefore, the size reduction of a particle. In some cases, however, the particle is only partially fractured and additional treatments are required to complete disassociation of the resulting fragments or sub-particles and, hence, the size reduction of the initial particle.

30 If the initial pressure pulse treatment of particles has been sufficient to break the particles to a desired resultant size, the flow of the treated dispersion can be directed by way of the block valve's setting out of the

machine. If additional treatments are required, the flow is directed to a filter chamber 12 which contains a screen across the path of the slurry flow. The screen acts to shear semi-fractured particles, which have not been fully fractured by the pressure pulse treatment, as they are rammed
5 against the surfaces of the screen. Particles which pass through the screen are unaffected by the shear and would remain semi-fractured.

The slurry flow then enters a baffled chamber 13, as illustrated schematically in Figure 3, which contains a series of baffle ring plates 54 along its interior. The slurry 18 enters the baffled chamber 13 through a
10 nozzle 51 which constricts the flow and, therefore, increases the fluid velocity through the chamber. The baffle plates 54 having protrusions 54A extend into the fluid flow further causing turbulence 55 to form within the flow. The action of the turbulence 55 and the impact of the particles 16 against protrusions 54A cause shear force to be created
15 within the particles. This causes particles not completely fractured by the pressure pulse treatment or the filter chamber to break into the desired smaller particles 14 which emerge from the baffle chamber in liquid vehicle 15.

In Figure 1, the baffled chamber 13 is shown after the filter
20 chamber 12. In other variations of the system, the baffled chamber may be placed before the filter chamber. Additionally, multiple filters and multiple baffled chambers may be employed to obtain a particular size reduction of a particular target material.

It should be noted from the above description that the process of
25 the present invention is believed to use the abrupt pressure change treatment to create a shock wave which fractures the particle. Pressure is used to draw the dispersion containing the target particles through the system shown in Figure 1, thus allowing virtually continuous size reduction of dispersed solid and semi-solid particulates.

30 By repeatedly recycling or passing the slurry through the system, the target particles can be reduced to the desired size. Recycling can be performed automatically using a closed-loop system or manually with open

system depicted in Figure 1. In manual application, pressure pulse treatment during the second and subsequent passes can significantly reduce the size of the particulates made during the first pass. The pressure level of subsequent passes may be increased, decreased, or
5 maintained constant, depending upon the amount of size reduction desired. When the particles have reached the desired size range, they may be filtered from the slurry or the slurry may be used as the final product containing the dispersed reduced sized particles.

In Figures 1 and 2, the inlet and outlet valves 9 and 10 may be any
10 valve system which effectively opens and closes in relation to the timing of the rise and falling of the piston 2. The valves may be mechanically, electrically, or pneumatically operated. The preferred embodiment simply uses spring-loaded check valves as illustrated in Figure 4. The inlet valve 9 allows the dispersion containing the dispersed particulates to enter the
15 compression chamber 17, and there become trapped between the inlet check valve 9 and the outlet check valve 10. The tension is adjusted, especially on the outlet check valve's spring 10, to keep the slurry confined within the chamber for a longer period of time, assisting in the maintenance of the pressure shock wave generated by the piston 2
20 striking the slurry. If repeated piston strokes on the same portion of the dispersion are desired, manually-operated valves, rather than the spring-loaded valves of the preferred embodiment, may be implemented.

EXAMPLE 1

A pressure pulse particle micronizing device was constructed
25 according to the present invention as shown in Figure 5. A reservoir 8 is attached to a modified hydraulic pump 1. The pump used was model number SC10-500-8 supplied by SC Hydraulics Corp. An Exit pipe 20 is attached to the outlet check valve 10 of the system. Attached to the pump 1 is an air filter 21 through which compressed air enters the system.
30 The air then passes through a pressure control regulator 4, a line oiler 5 and into the 1/4 turn air valve 6A where the flow into the air motor 3 of the pump 1 is controlled. The 1/4 turn air valve 6A has a dial 6B on its

face, marked with gradients from 0 to 9, which in this case correlates to 0 to 90 degrees. The valve is closed when the control level 6C is at 0 degrees and is fully open at setting 9, or 90 degrees. At setting zero the pump 1 is at rest. At setting 9 on the dial the pump is running at maximum speed.

The flow tube 20 in Figure 5 allows the pressure treated slurry 18 to exit the pump's compression chamber 17 once the outlet check valve 10 opens. Tube 20 may direct the slurry from the system immediately upon leaving the compression chamber 17, if additional treatment is not desired.

However, as shown in Figure 5, the flow tube 20 leads into a block valve 11 which directs the flow to a filter chamber 13 containing a 35 micron screen insert. From there the flow travels through a baffled size chamber 12 with the final exit from the system coming immediately thereafter. In some experiments, multiple filters were employed along with multiple baffled size chambers. In some experiments the outflow particulate 14 of dispersion 18 is automatically recycled back to the reservoir 8 for repeated pressure treatments.

In Figure 4, the slurry flow enters the compression chamber 17 of the air pump 3 through a spring-loaded crack valve assembly consisting of an inlet check valve 9 and an outlet check valve 10. The tension of the springs of both check valves is adjusted to allow both to remain closed during the downward stroke of the piston 2. This confines the slurry within the compression chamber 17, causing an abrupt pressure change which, it is believed, generates a high pressure shock wave. In this test apparatus, a pressure was generated which was 110 times the gauge pressure indication. Therefore, when the gauge of the air regulator 4 of Figure 5 indicates an inlet air pressure to the pump of 60 psi, the geometry of the compression chamber and the dimensions of the piston 2 coupled with the force of the piston act to increase the instantaneous pressure within the compression chamber 17 by a factor of 110 times the gauge pressure indication to 6,600 psi. The pressure is applied for 1/37

seconds, while the slurry is in the compression chamber, before the next stroke of the piston evacuates the compression chamber and draws in another batch of slurry for treatment.

A slurry including a liquid medium of 50% hexane and 50% isopropanol is made by mixing 50 ml of each into a 1 liter beaker under mild agitation at ambient temperature. The particulate is 150 grams of Aspartame, supplied by Tosoh Canada Ltd., and is stirred mildly into the beaker of liquid medium to form the slurry or dispersion. The combination of hexane and isopropanol was chosen as the liquid medium because aspartame was only mildly soluble in this dispersion.

Referring to Figure 5, the dispersion of liquid medium and Aspartame is added to the reservoir 8, and the regulator 4 on the device is set to various pressure settings as indicated in the following Table 1. The inlet pressure setting for the initial test run was 60 psi.

TABLE 1

Size Reduction By Pressure - Tosoh Aspartame

	Size (Microns)	Percent Reduction	Pressure (PSI) *	Pressure (PSI) **
25	29.0	0	0	
	9.9	67.0	60	
	9.0	69.0	70	
	9.3	67.9	80	
	5.0	82.8		60
30	4.0	86.2		90

* Chamber 2 and 1

** Chamber 2

The Aspartame, solvent carrier dispersion was applied to the reservoir 8 of the device illustrated in Figure 5.

At setting 0 the machine is not in operation. The size range indicated of 29 microns is the average starting size of the Aspartame

particle. The designation 2 indicates that baffle chamber 13 of size number 2 was used. A 35 micron filter screen is used in the filter chamber 12.

Size number 2 of baffle chamber 13 includes only 4 baffle rings in a chamber approximately 4 inches long corresponding to the design shown in Figure 3. Baffle chamber number 1 consists of 8 baffle rings in a chamber approximately 4 inches long and corresponding to the design shown in Figure 3. The turbulence in chamber number 1 is greater than that of chamber number 2.

Where the designation (2&1) is used, both size number 2 and size number 1 baffle chambers are used in the same application. The size number 1 chamber was positioned downstream of size number 2 chamber in an otherwise identical configuration. Referring to Figure 5, the design includes a filter chamber 12 and, where one baffle chamber is indicated in the figure, two chambers were used instead of one, with size chamber number 2 fluidly connected to filter chamber 12 and size chamber number 1 connected directly behind size chamber number 2 in a series flow configuration. The outflow from chamber number 1 exits the machine. In this series flow configuration the chamber with the lesser turbulence acts to shear particles flowing through that chamber moderately breaking the particles into smaller sized bits. Chamber 1, with the higher turbulence factor, is intended to shear those particles processed by chamber number 2 into even smaller sizes.

The placement of the size chambers could be reversed and even placed ahead of the filter chamber.

In the experiment illustrated by the results shown in Table 1, the aspartame slurry was passed through the machine only once. A size scan study of raw, untreated aspartame (unsuspended) was first performed. Size reduction measurements of the particles after each treatment of varying pressure settings and baffle chamber configurations were then performed by scanning the treated slurry. All measurements were taken using a laser diffraction size determination device supplied by Leeds and

Northrup and known as Microtrac. At the conclusion of each test run, the pressure micronizing device of the present invention was cleansed of all residue and flushed with water. Size scans were also conducted on the water flush liquid to verify that no sediment remained in the machine from the previous runs, to avoid contaminating the next test.

The test runs consisted of a single pass of the aspartame slurry through the test machine as illustrated in Figure 4, with a speed dial setting at 3 throughout all of the tests, with varying pressure settings on the inlet regulator and using different size baffle chamber configurations.

At a setting of 60 psi using both baffle chambers (size number 1 and 2), the size was reduced on just one pass of the aspartame slurry through the machine to just 9.9 microns. On the second test run the setting was changed to 70 psi, using the same starting aspartame slurry, and the size of the aspartame particles was reduced to 9.0 microns. At the 80 psi setting, the size reduction was 9.3 microns. This indicates that the pump is operating close to its maximum pressure rating.

Tests were then conducted with only one baffle chamber. At 60 psi (2), which employs a regulator pressure setting of 60 psi and only size chamber number 2 (the chamber with the milder turbulence baffle ring configuration), the aspartame was reduced in size to 5 microns, indicating superior performance compared with the 2 baffle chambers in series configuration. Further examination indicates the average particle size reduction is greater with the use of a single particular size chamber, however, the overall size range is narrowed with the use of the 2 chambers in series configuration. At 90 psi the size was reduced to just 4 microns from a starting size of 29 microns.

Table 1 also lists the percentage of size reduction obtained at each setting for each test run.

Table 2, shown below, illustrates the effects of a recycling of the aspartame slurry through the system as configured in Figure 5 using size chambers 2&1 and a pressure of 60 psi. The first pass reduced the aspartame average particle size to just 9.9 microns. This slurry was then

re-cycled through the machine for a second pass at the same pressure setting resulting in further particle reduction to 3.5 microns. On the third pass, the size was further reduced to just 1.4 microns. Accordingly, the apparatus can further reduce the size of particles by recycling the slurry through the machine more than once.

TABLE 2

Size Reduction Upon Recycling - Tosoh Aspartame

Pass #	Size (Microns)	Pressure (PSI)
0	29.0	0
1	9.9	60
2	3.5	60
3	1.4	60

EXAMPLE 2

Following Table 3 shows the results of tests using the configuration as illustrated in Figure 5 using the two chamber configuration at various regulator gauge pressure settings, with the speed dial set at a constant level 3 through the test runs. Each setting used a new batch of slurry including by weight 83% distilled water and 15% antimony oxide. The antimony oxide was supplied by Asarco Inc. The antimony oxide particles had a starting average particle size of 5 microns. At each pressure setting the size was reduced with the greatest size reduction occurring at the highest pressure setting. Table 3 also lists the percentage of size reduction for antimony oxide. It should be noted that antimony oxide is a harder substance than is aspartame.

TABLE 3

Size Reduction of Asarco Antimony Oxide
(Filter and Baffle Chambers Used)

	<u>Size (Microns)</u>	<u>Percent Reduction</u>	<u>Pressure (PSI)</u>
5	4.9	0	0
10	3.8	22.4	60
	3.8	22.4	70
	3.4	30.6	80

15 Table 4, shown below, lists the effects of size reduction on the antimony oxide when the machine is configured with no filter or size chamber attachments, as otherwise shown in Figure 5, at gauge pressures of 60, 70, and 80 psi. In each case, the size of the particles was reduced, although not as much as when the filter and baffle chambers
20 were used. The results show that the abrupt pressure change caused by the piston alone will break the particles into smaller pieces, although the efficiency is improved by the addition of the baffle and filter chambers.

TABLE 4

Size Reduction of Asarco Antimony Oxide
(No Filter or Size Chambers)

	<u>Size (Microns)</u>	<u>Percent Reduction</u>	<u>Pressure (PSI)</u>
25	5.0	0	0
30	4.5	10.0	60
	4.0	20.0	70
35	3.5	30.0	80

While the invention has been described with respect to specific
embodiments, it is to be understood that many variations of the present
40 invention are possible. The embodiments of the invention described herein
are intended to be illustrative, and are not intended to be limiting as to the
scope of the invention as defined by the claims appended hereto.

- Variations of the apparatus have been shown wherein an abrupt pressure change is generated by a piston pump apparatus with locking check valves, acting alone or in conjunction with a filter screen chamber and/or baffled chambers which induce turbulence to aid in the size
- 5 reduction of particles which are dispersed within a liquid medium. It may be possible to substitute a gas for the liquid medium and other pressure application means for the piston. Multiple pistons may be substituted for the single piston shown in the preferred embodiment.

We claim:

1. A method of reducing the size of particles dispersed in a fluid medium which comprises: treating the dispersion by subjecting it to an abrupt pressure change while the dispersion is confined in a compression chamber, thereby causing the particles to be fractured and to break apart along the interface of the fracture.
2. The method of claim 1 which includes the additional steps of repeatedly and intermittently receiving and evacuating portions of the dispersion into and from the compression chamber, thereby subjecting successive portions of the dispersion to the abrupt pressure change.
3. The method of claim 2 wherein the dispersion is confined within the compression chamber by a pair of valves, and wherein the abrupt pressure change is caused by a piston impacting the confined portion of the dispersion in the compression chamber.
4. The method of claim 1 which further includes the step of passing the treated dispersion through a screen.
5. The method of claim 2 which further includes the step of passing the treated dispersion through a screen.
6. The method of claim 3 which further includes the step of passing the treated dispersion through a screen.
7. The method of claim 1 which further includes the step of passing the treated dispersion through a baffled chamber.
8. The method of claim 2 which further includes the step of passing the treated dispersion through a baffled chamber.
9. The method of claim 3 which further includes the step of passing the treated dispersion through a baffled chamber.
10. An apparatus for reducing the size of particles dispersed within a fluid which comprises: a compression chamber, having an inlet valve and an outlet valve, adapted to contain the dispersion; and impact means, operatively connected to said compression chamber which acts on the dispersion causing it to be subjected to an abrupt pressure change,

thereby causing the particles to be fractured and to break apart along the interface of the fracture.

11. The apparatus of claim 10 which further includes means for activating said valves so that portions of the dispersion are intermittently
5 and successively injected into and withdrawn from said compression chamber, thereby subjecting successive portions of the dispersion to an abrupt pressure change.

12. The apparatus of claim 10 which further includes a screen
10 through which the dispersion containing the fractured particles is passed through.

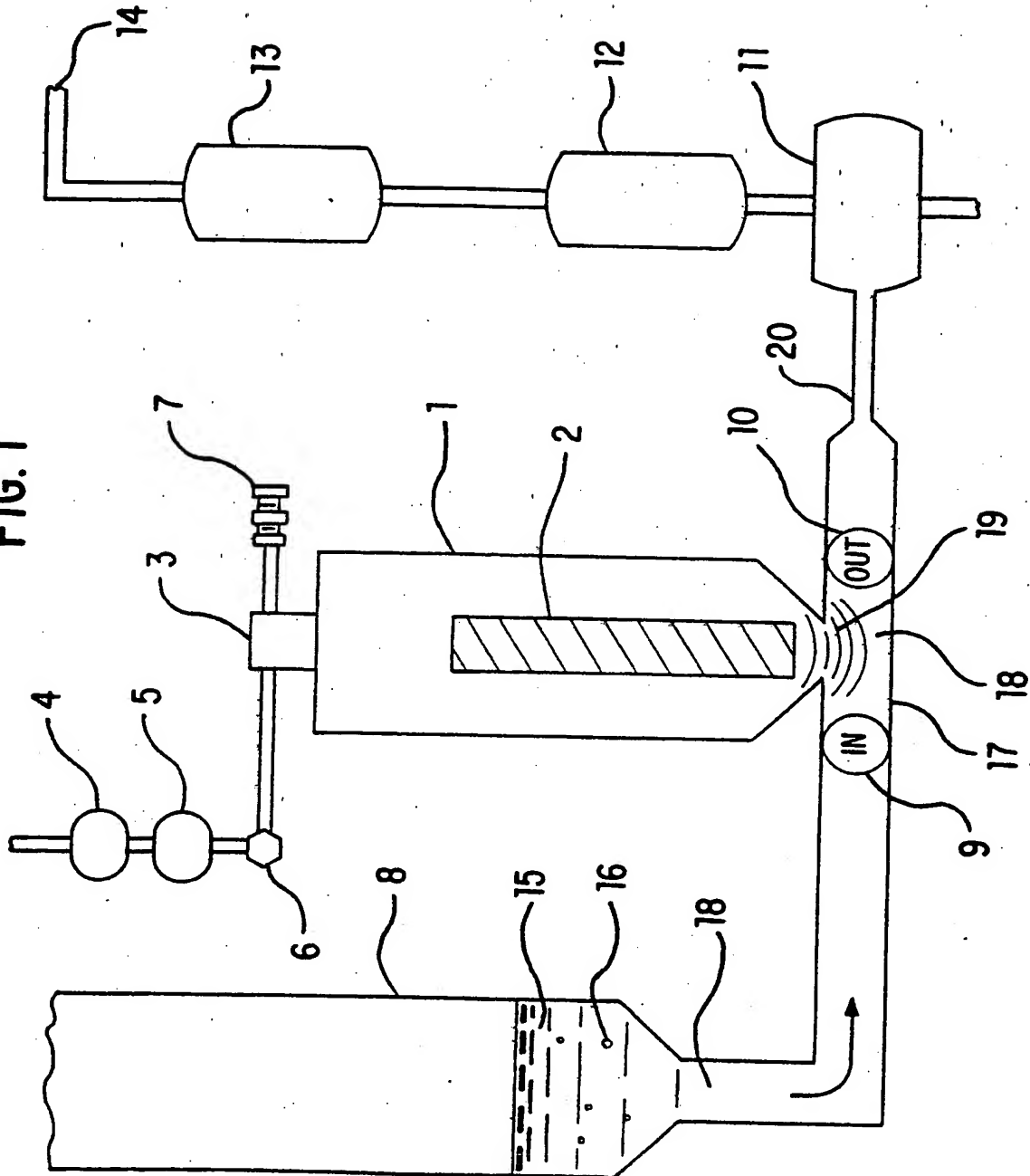
13. The apparatus of claim 11 which further includes a screen through which the dispersion containing the fractured particles is passed through.

14. The apparatus of claim 10 which further includes a baffle
15 chamber through which the dispersion containing the fractured particles is passed through.

15. The apparatus of claim 11 which further includes a baffle chamber through which the dispersion containing the fractured particles is passed through.

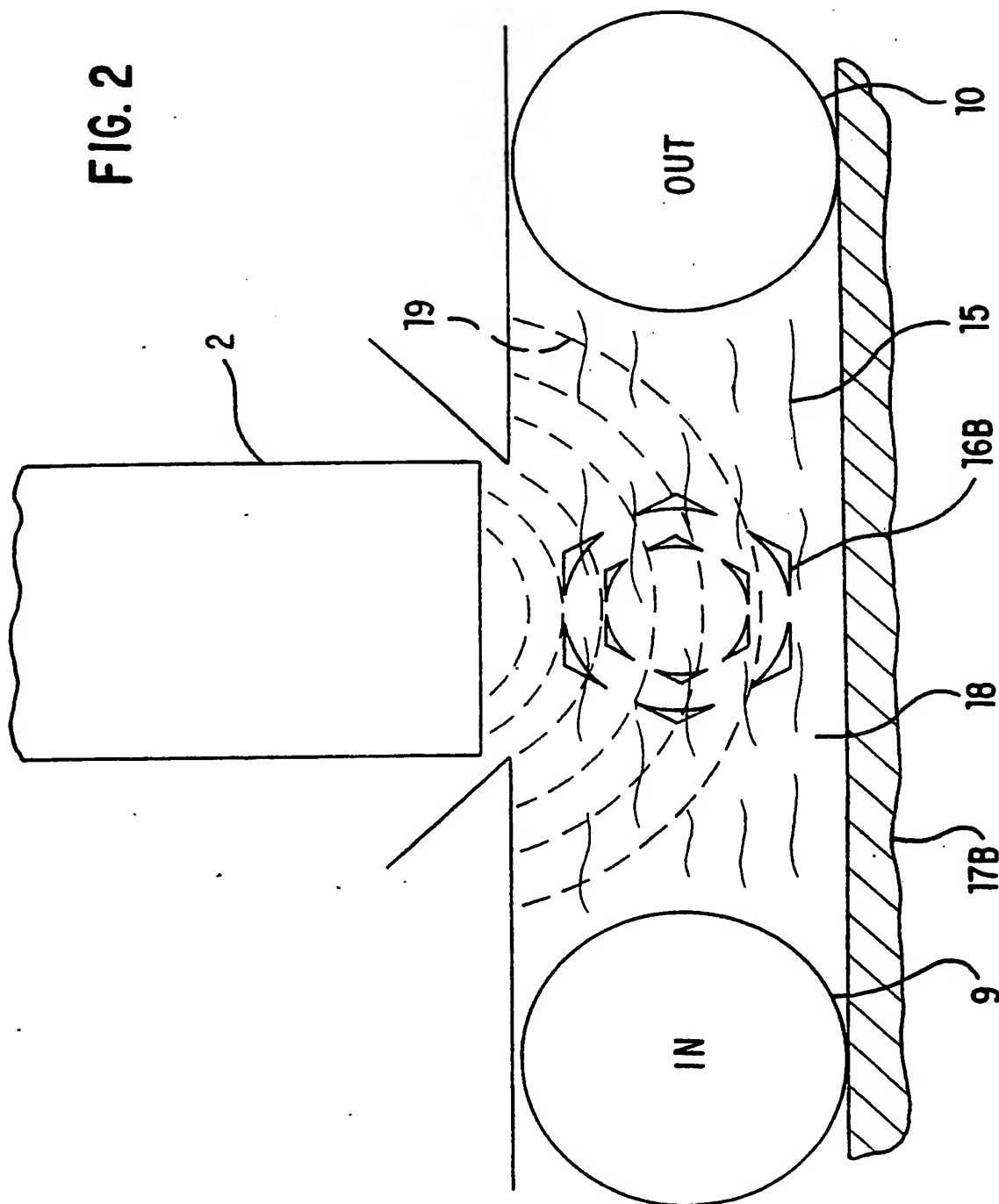
1/5

FIG. 1

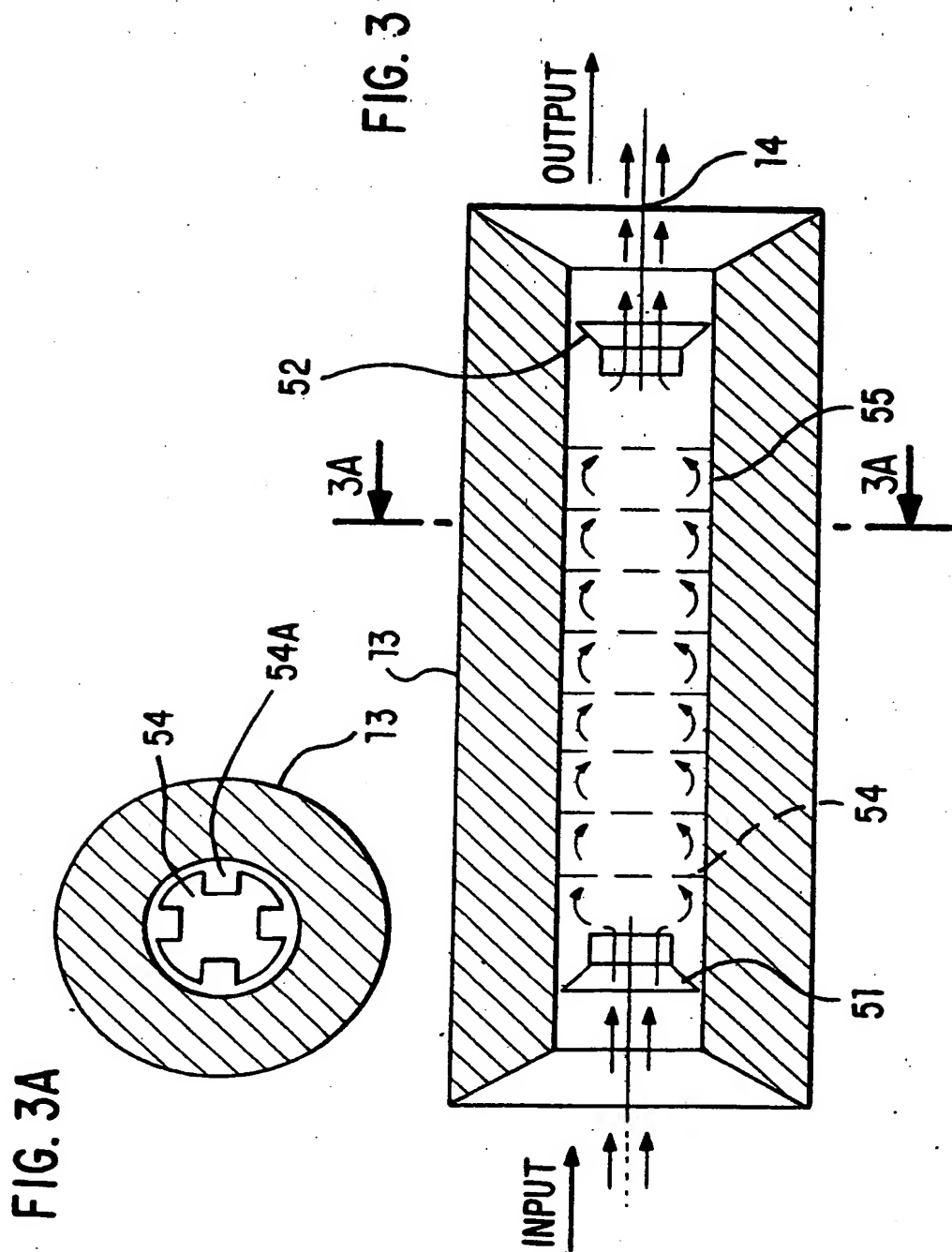


2/5

FIG. 2



3/5



4/5

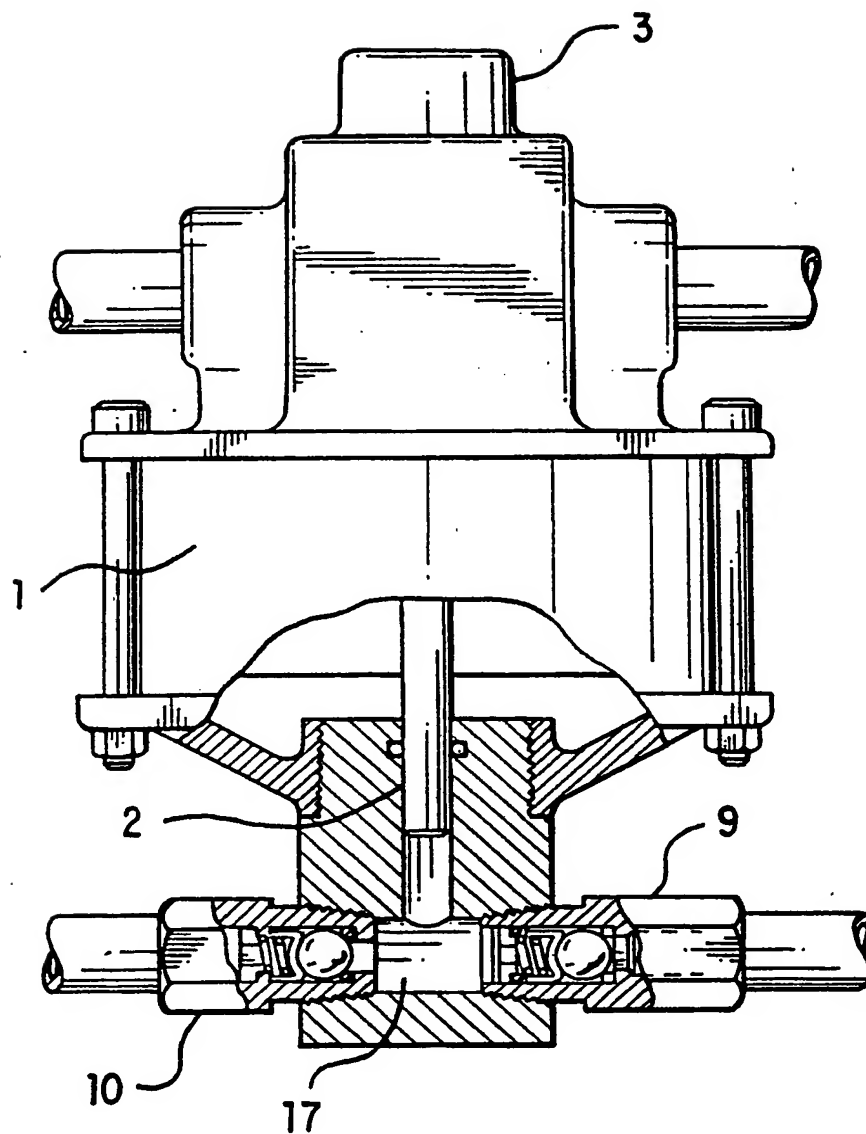
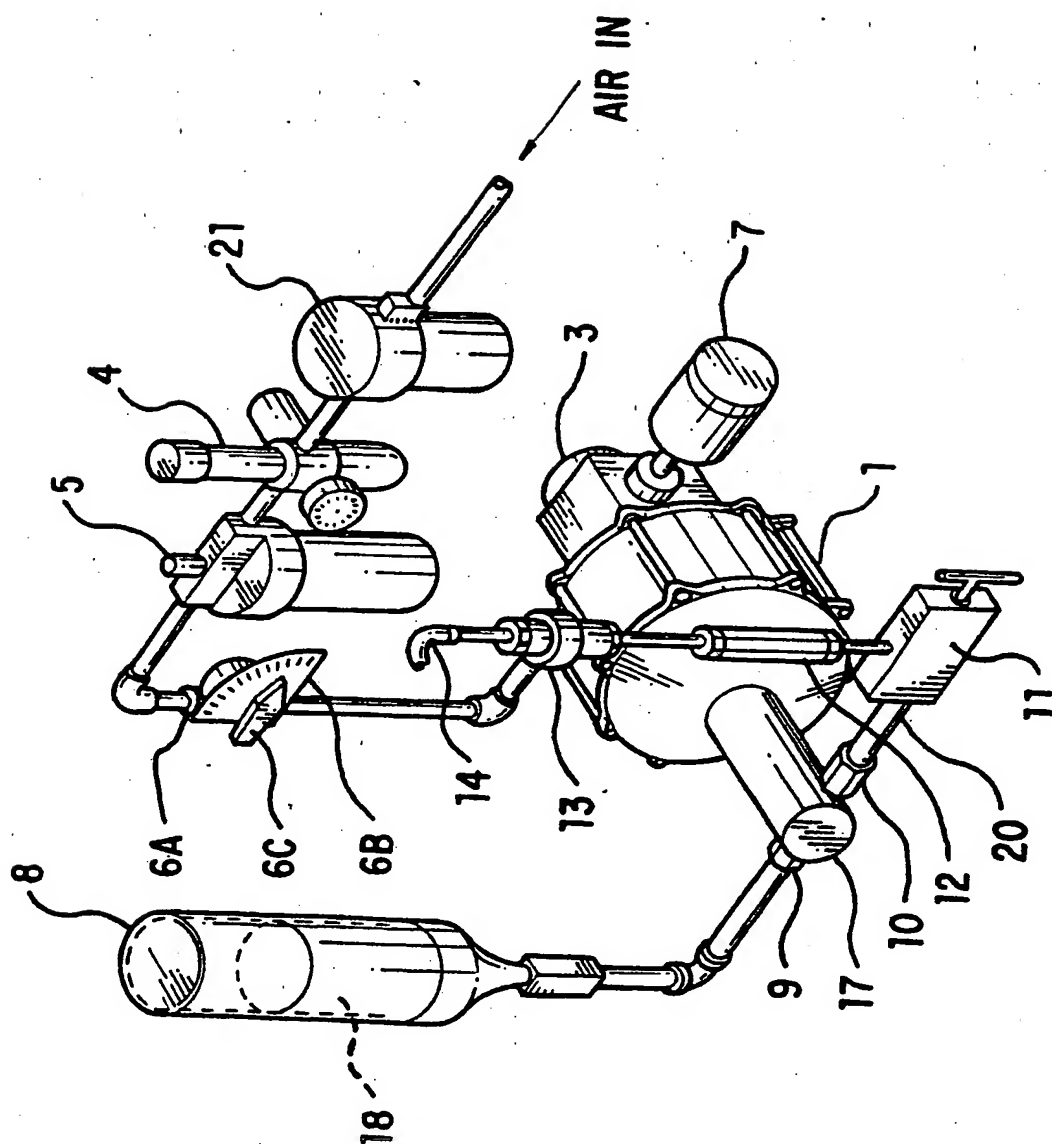


FIG. 4

5/5



INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/09089

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all)		
According to International Patent Classification (IPC) or to both National Classification and IPC: IPC (5): BO2C 23/36 U.S.Cl.: 241/1		
II. FIELDS SEARCHED		
Minimum Documentation Searched		
Classification System	Classification Symbols	
U.S.	241/1, 21, 24, 29, 152A, 301	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched		
III. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of Document, ** with indication, where appropriate, of the relevant passages *2	Relevant to Claim No. *3
X	US, A, 1,230,013 (MORWITZ) 12 June 1917 See column 2, lines 77-89 and Figure 2.	1 and 10
X	EPO 0013094 (LLOYD) 09 July 1980 See pg. 7, lines 1-39 and Figure 1.	1-6 & 10-13
Y	US, A, 3,207,447 (WHITHAM) 21 September 1965	7-9, 14&15
A	SU - 716-591 (POVOLOTSKII) 08 August 1977	1 and 10
A	US, A, 4,053,110 (SCHALKOWSKY ET AL.) 11 October 1977	1 and 10
A	US, A, 4,156,593 (TARPLEY, JR.) 29 May 1979	1 - 15
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: *4</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search		Date of Mailing of this International Search Report
12 March 1992		31 MAR 1992
International Searching Authority		Signature of Authorized Officer
ISA/US		 John M. Husar

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record.**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☐ FADED TEXT OR DRAWING
- ☐ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☒ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☒ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☐ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

THIS PAGE BLANK (USPTO)